

CLAIMS

1. Amorphous 5-chloro-N-[(1S,2R)-3-[3R,4S]-3,4-dihydroxy-1-pyrrolidinyl]-2-hydroxy-3-oxo-1-(phenylmethyl)propyl]-1H-indole-2-carboxamide.

5

2. A crystal form of anhydrous 5-chloro-N-[(1S,2R)-3-[3R,4S]-3,4-dihydroxy-1-pyrrolidinyl]-2-hydroxy-3-oxo-1-(phenylmethyl)propyl]-1H-indole-2-carboxamide (Form A), which crystal form comprises characteristic high intensity X-ray diffraction peaks at diffraction angles (2-theta) of about 12.73, 16.82, 17.67, 20.24, 20.56, 20.96, 21.41, 25.61 and 25.85, and X-ray diffraction d-spacings (Å) of about 6.95, 5.27, 5.02, 4.38, 4.32, 4.23, 4.15, 3.48, and 3.44, respectively.

10

3. A crystal form according to claim 2, which crystal form further comprises characteristic diffraction peaks at diffraction angles (2-theta), X-ray diffraction d-spacings (Å), and intensities (I) of about:

15

| Angle | d-value (Å) | I | Angle | d-value (Å) | I | Angle | d-value (Å) | I |
|---------|-------------|-------|---------|-------------|------|---------|-------------|------|
| 2-Theta | | | 2-Theta | | | 2-Theta | | |
| 6.48 | 13.62 | 20.9 | 22.54 | 3.94 | 5.2 | 30.46 | 2.93 | 6.0 |
| 12.73 | 6.95 | 80.4 | 24.44 | 3.64 | 9.8 | 31.10 | 2.87 | 8.9 |
| 14.11 | 6.27 | 6.7 | 24.72 | 3.60 | 11.9 | 31.44 | 2.84 | 6.9 |
| 16.82 | 5.27 | 34.4 | 25.01 | 3.56 | 14.0 | 32.39 | 2.76 | 10.1 |
| 17.67 | 5.02 | 35.0 | 25.61 | 3.48 | 32.4 | 33.24 | 2.69 | 6.7 |
| 18.69 | 4.74 | 12.3 | 25.85 | 3.44 | 30.4 | 35.76 | 2.51 | 5.0 |
| 19.12 | 4.64 | 8.0 | 26.76 | 3.33 | 23.2 | 36.09 | 2.49 | 6.6 |
| 20.24 | 4.38 | 32.8 | 27.47 | 3.24 | 7.7 | 36.87 | 2.44 | 6.2 |
| 20.56 | 4.32 | 100.0 | 28.08 | 3.18 | 6.5 | 37.12 | 2.42 | 6.8 |
| 20.96 | 4.23 | 30.1 | 28.55 | 3.12 | 13.8 | 38.85 | 2.32 | 7.3 |
| 21.41 | 4.15 | 74.7 | 30.04 | 2.97 | 5.3 | | | |

4. A crystal form of 5-chloro-N-[(1S,2R)-3-[3R,4S]-3,4-dihydroxy-1-pyrrolidinyl]-2-hydroxy-3-oxo-1-(phenylmethyl)propyl]-1H-indole-2-carboxamide, mono-ethanolate (Form C), which crystal form comprises characteristic high intensity X-ray diffraction peaks at diffraction angles (2-theta) of about 6.26, 8.70, 18.46, 19.22, 21.24, 23.27, 23.62, 23.87, and X-ray diffraction d-spacings of about 14.12, 10.16, 4.80, 4.61, 4.18, 3.82, 3.76, and 3.72, respectively.

20

5. A crystal form according to claim 4, which crystal form further comprises characteristic diffraction peaks at diffraction angles (2-theta), X-ray diffraction d-spacings (Å), and intensities (I) of about:

| Angle | d-value (Å) | I | Angle | d-value (Å) | I | Angle | d-value (Å) | I |
|---------|-------------|------|---------|-------------|-------|---------|-------------|------|
| 2-Theta | | | 2-Theta | | | 2-Theta | | |
| 4.36 | 20.25 | 8.6 | 19.75 | 4.49 | 18.9 | 29.13 | 3.06 | 7.5 |
| 6.26 | 14.12 | 46.8 | 20.71 | 4.28 | 28.6 | 29.63 | 3.01 | 12.4 |
| 8.70 | 10.16 | 95.0 | 21.24 | 4.18 | 100.0 | 30.23 | 2.95 | 16.0 |
| 10.40 | 8.50 | 10.7 | 21.77 | 4.08 | 20.5 | 30.61 | 2.92 | 12.0 |
| 10.99 | 8.05 | 37.3 | 22.14 | 4.01 | 9.0 | 31.00 | 2.88 | 15.8 |
| 12.56 | 7.04 | 33.8 | 23.27 | 3.82 | 75.5 | 32.49 | 2.75 | 13.3 |
| 15.57 | 5.69 | 28.6 | 23.62 | 3.76 | 55.3 | 33.26 | 2.69 | 8.2 |
| 16.06 | 5.52 | 10.9 | 23.87 | 3.72 | 52.8 | 33.98 | 2.64 | 15.2 |
| 16.41 | 5.40 | 14.6 | 24.33 | 3.65 | 10.5 | 34.78 | 2.58 | 8.5 |
| 16.76 | 5.29 | 8.5 | 25.61 | 3.48 | 8.4 | 35.46 | 2.53 | 12.6 |
| 17.18 | 5.16 | 15.5 | 26.10 | 3.41 | 33.1 | 36.00 | 2.49 | 8.8 |
| 17.49 | 5.07 | 9.4 | 26.60 | 3.35 | 21.3 | 37.36 | 2.40 | 8.0 |
| 18.46 | 4.80 | 94.1 | 26.88 | 3.31 | 15.4 | 39.03 | 2.31 | 6.7 |
| 19.22 | 4.61 | 46.1 | 28.16 | 3.17 | 19.4 | | | |

- 5 6. A crystal form of 5-chloro-N-[(1S,2R)-3-[3R,4S]-3,4-dihydroxy-1-pyrrolidinyl]-2-hydroxy-3-oxo-1-(phenylmethyl)propyl]-1H-indole-2-carboxamide, dihydrate (Form D), which crystal form comprises characteristic high intensity X-ray diffraction peaks at diffraction angles (2-theta) at about 4.90, 8.53, 14.38, 18.13, 21.74, 22.23, 22.46, 22.97 and 23.45, and X-ray diffraction d-spacings of about 18.02, 10.36, 6.15, 4.89, 4.08, 4.00, 3.96, 3.87, and 3.79, respectively.

7. A crystal form according to claim 6, which crystal form further comprises characteristic diffraction peaks at diffraction angles (2-theta), X-ray diffraction d-spacings (Å), and intensities (I) of about:

| Angle | d-value (Å) | I | Angle | d-value (Å) | I | Angle | d-value (Å) | I |
|---------|-------------|------|---------|-------------|------|---------|-------------|------|
| 2-Theta | | | 2-Theta | | | 2-Theta | | |
| 4.90 | 18.02 | 60.1 | 18.61 | 4.76 | 44.4 | 27.46 | 3.24 | 32.0 |
| 6.61 | 13.35 | 11.6 | 19.74 | 4.49 | 29.9 | 28.04 | 3.18 | 24.2 |
| 8.53 | 10.36 | 71.8 | 20.08 | 4.42 | 55.1 | 28.94 | 3.08 | 17.2 |
| 9.75 | 9.07 | 44.2 | 20.56 | 4.32 | 20.2 | 29.46 | 3.03 | 14.6 |
| 13.32 | 6.64 | 19.3 | 21.26 | 4.18 | 30.7 | 30.23 | 2.95 | 14.0 |
| 13.64 | 6.49 | 25.7 | 21.74 | 4.08 | 75.7 | 30.70 | 2.91 | 12.5 |
| 13.86 | 6.39 | 37.8 | 22.23 | 4.00 | 69.6 | 30.94 | 2.89 | 15.3 |
| 14.38 | 6.15 | 58.7 | 22.46 | 3.96 | 65.9 | 31.24 | 2.86 | 11.8 |

| | | | | | | | | |
|-------|------|------|-------|------|-------|-------|------|------|
| 14.72 | 6.01 | 12.1 | 22.97 | 3.87 | 100.0 | 32.03 | 2.79 | 10.1 |
| 14.98 | 5.91 | 12.1 | 23.45 | 3.79 | 57.7 | 32.48 | 2.75 | 13.3 |
| 15.32 | 5.78 | 12.4 | 24.22 | 3.67 | 12.7 | 32.83 | 2.73 | 14.4 |
| 15.76 | 5.62 | 16.1 | 24.72 | 3.60 | 11.8 | 33.57 | 2.67 | 12.5 |
| 15.96 | 5.55 | 22.5 | 25.63 | 3.47 | 13.3 | 35.23 | 2.55 | 10.4 |
| 17.55 | 5.05 | 38.2 | 26.28 | 3.39 | 40.8 | 35.72 | 2.51 | 11.4 |
| 18.13 | 4.89 | 57.8 | 26.83 | 3.32 | 33.7 | | | |

8. A crystal form of 5-chloro-N-[(1S,2R)-3-[3R,4S]-3,4-dihydroxy-1-pyrrolidinyl]-2-hydroxy-3-oxo-1-(phenylmethyl)propyl]-1H-indole-2-carboxamide, hemi-ethanolate (Form E), which crystal form comprises characteristic high intensity X-ray diffraction peaks at diffraction angles (2-theta) at about 11.97, 16.80, 20.07, 21.06, 22.00, 23.89, and 26.27, and X-ray diffraction d-spacings of about 7.39, 5.27, 4.42, 4.22, 4.04, 3.72, and 3.39, respectively.

9. A crystal form according to claim 8, which crystal form further comprises characteristic diffraction peaks at diffraction angles (2-theta), X-ray diffraction d-spacings (Å), and intensities (I) of about:

| Angle | d-value (Å) | I | Angle | d-value (Å) | I | Angle | d-value (Å) | I |
|---------|-------------|------|---------|-------------|-------|---------|-------------|------|
| 2-Theta | | | 2-Theta | | | 2-Theta | | |
| 4.75 | 18.60 | 7.2 | 17.88 | 4.96 | 19.5 | 26.90 | 3.31 | 46.8 |
| 6.57 | 13.45 | 10.4 | 18.65 | 4.75 | 32.7 | 27.97 | 3.19 | 22.5 |
| 6.82 | 12.94 | 9.5 | 19.62 | 4.52 | 34.7 | 28.66 | 3.11 | 18.4 |
| 7.53 | 11.74 | 25.2 | 20.07 | 4.42 | 52.8 | 29.98 | 2.98 | 17.8 |
| 9.81 | 9.01 | 5.8 | 21.06 | 4.22 | 96.4 | 31.19 | 2.87 | 30.3 |
| 10.47 | 8.44 | 7.8 | 22.00 | 4.04 | 100.0 | 32.60 | 2.74 | 17.4 |
| 11.97 | 7.39 | 75.1 | 22.72 | 3.91 | 27.7 | 36.72 | 2.45 | 12.7 |
| 13.00 | 6.80 | 9.8 | 23.89 | 3.72 | 50.1 | 37.03 | 2.43 | 12.2 |
| 13.54 | 6.54 | 9.9 | 24.86 | 3.58 | 26.6 | | | |
| 16.80 | 5.27 | 64.3 | 26.27 | 3.39 | 65.9 | | | |

10. A crystal form of 5-chloro-N-[(1S,2R)-3-[3R,4S]-3,4-dihydroxy-1-pyrrolidinyl]-2-hydroxy-3-oxo-1-(phenylmethyl)propyl]-1H-indole-2-carboxamide, monohydrate (Form F), which crystal form comprises characteristic high intensity X-ray diffraction peaks at diffraction angles (2-theta) at about 4.30, 6.07, 9.67, 13.66, 17.88, 20.09, and 21.88, and X-ray diffraction d-spacings of about 20.54, 14.54, 9.14, 6.48, 4.96, 4.42, and 4.06, respectively.

11. A crystal form according to claim 10, which crystal form further comprises characteristic diffraction peaks at diffraction angles (2-theta), X-ray diffraction d-spacings (Å), and intensities (I) of about:

| Angle | d-value (Å) | I | Angle | d-value (Å) | I | Angle | d-value (Å) | I |
|---------|-------------|-------|---------|-------------|------|---------|-------------|------|
| 2-Theta | | | 2-Theta | | | 2-Theta | | |
| 4.30 | 20.54 | 100.0 | 22.62 | 3.93 | 16.7 | 31.07 | 2.88 | 10.9 |
| 6.07 | 14.54 | 66.1 | 24.28 | 3.66 | 14.0 | 31.92 | 2.80 | 9.3 |
| 8.61 | 10.26 | 26.3 | 24.54 | 3.62 | 21.8 | 32.25 | 2.77 | 12.2 |
| 9.67 | 9.14 | 47.2 | 25.03 | 3.56 | 21.6 | 32.86 | 2.72 | 13.5 |
| 13.09 | 6.76 | 5.4 | 25.32 | 3.51 | 16.3 | 33.88 | 2.64 | 5.8 |
| 13.66 | 6.48 | 40.6 | 25.69 | 3.46 | 17.1 | 34.61 | 2.59 | 6.9 |
| 15.86 | 5.58 | 17.8 | 26.20 | 3.40 | 11.4 | 35.19 | 2.55 | 8.6 |
| 17.04 | 5.20 | 8.2 | 26.63 | 3.34 | 11.0 | 35.81 | 2.51 | 7.0 |
| 17.88 | 4.96 | 98.9 | 27.55 | 3.23 | 13.0 | 36.20 | 2.48 | 5.3 |
| 18.54 | 4.78 | 30.7 | 28.18 | 3.16 | 11.4 | 37.03 | 2.43 | 7.5 |
| 19.14 | 4.63 | 37.3 | 29.00 | 3.08 | 10.4 | 37.98 | 2.37 | 6.8 |
| 20.09 | 4.42 | 44.7 | 29.16 | 3.06 | 12.0 | 39.24 | 2.29 | 5.2 |
| 21.47 | 4.14 | 21.3 | 29.40 | 3.04 | 11.3 | 39.69 | 2.27 | 5.8 |
| 21.88 | 4.06 | 53.0 | 30.36 | 2.94 | 5.5 | | | |

- 5 12. A crystal form of anhydrous 5-chloro-N-[(1S,2R)-3-[3R,4S]-3,4-dihydroxy-1-pyrrolidinyl]-2-hydroxy-3-oxo-1-(phenylmethyl)propyl]-1H-indole-2-carboxamide (Form G), crystal form comprises characteristic high intensity X-ray diffraction peaks at diffraction angles (2-theta) at about 18.2, 20.1, 24.3, 25.2, and 26.3, and X-ray diffraction d-spacings, expressed in Å, of about 4.9, 4.4, 3.7, 3.5, and 3.4, respectively.
- 10

13. A crystal form according to claim 12, which crystal form further comprises characteristic diffraction peaks at diffraction angles (2-theta), X-ray diffraction d-spacings (Å), and intensities (I) of about:

| Angle | d-value (Å) | I | Angle | d-value (Å) | I | Angle | d-value (Å) | I |
|---------|-------------|-------|---------|-------------|------|---------|-------------|-----|
| 2-Theta | | | 2-Theta | | | 2-Theta | | |
| 16.2 | 5.5 | 5.4 | 26.3 | 3.4 | 38.6 | 34.8 | 2.6 | 2.4 |
| 17.2 | 5.1 | 2.8 | 26.9 | 3.3 | 3.6 | 35.2 | 2.5 | 3.2 |
| 18.2 | 4.9 | 8.6 | 27.9 | 3.2 | 6.4 | 35.5 | 2.5 | 2.7 |
| 19.7 | 4.5 | 4.1 | 29.4 | 3.0 | 2.3 | 35.9 | 2.5 | 2.7 |
| 20.1 | 4.4 | 100.0 | 29.8 | 3.0 | 3.9 | 36.6 | 2.4 | 3.6 |
| 20.8 | 4.3 | 5.8 | 30.8 | 2.9 | 2.9 | 38.4 | 2.3 | 3.7 |
| 23.7 | 3.7 | 2.5 | 31.6 | 2.8 | 3.5 | 38.8 | 2.3 | 3.3 |
| 24.3 | 3.7 | 12.1 | 31.9 | 2.8 | 3.8 | 39.8 | 2.3 | 3.1 |

| | | | | | | | | |
|------|-----|-----|------|-----|-----|--|--|--|
| 25.2 | 3.5 | 8.9 | 32.7 | 2.7 | 3.7 | | | |
|------|-----|-----|------|-----|-----|--|--|--|

14. A process for preparing anhydrous 5-chloro-N-[(1S,2R)-3-[3R,4S]-3,4-dihydroxy-1-pyrrolidinyl]-2-hydroxy-3-oxo-1-(phenylmethyl)propyl]-1H-indole-2-carboxamide (Form A), which process comprises azotropically distilling a solution prepared from 5-chloro-N-[(1S,2R)-3-[3R,4S]-3,4-dihydroxy-1-pyrrolidinyl]-2-hydroxy-3-oxo-1-(phenylmethyl)propyl]-1H-indole-2-carboxamide, dihydrate (Form D), in an aprotic solvent to substantially remove said water of hydration and crystallize said Form A crystal form.
15. A process for preparing 5-chloro-N-[(1S,2R)-3-[3R,4S]-3,4-dihydroxy-1-pyrrolidinyl]-2-hydroxy-3-oxo-1-(phenylmethyl)propyl]-1H-indole-2-carboxamide, dihydrate (Form D), which process comprises the steps of:
 - (a) adding water to a suspension of anhydrous 5-chloro-N-[(1S,2R)-3-[3R,4S]-3,4-dihydroxy-1-pyrrolidinyl]-2-hydroxy-3-oxo-1-(phenylmethyl)propyl]-1H-indole-2-carboxamide (Form A), or anhydrous 5-chloro-N-[(1S,2R)-3-[3R,4S]-3,4-dihydroxy-1-pyrrolidinyl]-2-hydroxy-3-oxo-1-(phenylmethyl)propyl]-1H-indole-2-carboxamide (Form B) crystal forms in an aprotic solvent such that a water content of between about 5% and about 10% is achieved;
 - (b) heating said suspension to substantially dissolve said Form A or said Form B crystal form to form a solution; and
 - (c) diluting said solution with a non-polar, anti-solvent to crystallize said Form D crystal form.
16. A pharmaceutical composition comprising a Form A crystal form of anhydrous 5-chloro-N-[(1S,2R)-3-[3R,4S]-3,4-dihydroxy-1-pyrrolidinyl]-2-hydroxy-3-oxo-1-(phenylmethyl)propyl]-1H-indole-2-carboxamide, or a Form D crystal form of 5-chloro-N-[(1S,2R)-3-[3R,4S]-3,4-dihydroxy-1-pyrrolidinyl]-2-hydroxy-3-oxo-1-(phenylmethyl)propyl]-1H-indole-2-carboxamide, dihydrate, and a pharmaceutically acceptable carrier, vehicle, or diluent.
17. A method of treating a glycogen phosphorylase dependent disease or condition which method comprises administering to a mammal in need of such treatment a Form A crystal form of anhydrous 5-chloro-N-[(1S,2R)-3-[3R,4S]-3,4-dihydroxy-1-

pyrrolidinyl]-2-hydroxy-3-oxo-1-(phenylmethyl)propyl]-1H-indole-2-carboxamide, a Form D crystal form of 5-chloro-N-[(1S,2R)-3-[3R,4S]-3,4-dihydroxy-1-pyrrolidinyl]-2-hydroxy-3-oxo-1-(phenylmethyl)propyl]-1H-indole-2-carboxamide, dihydrate, or a pharmaceutical composition comprising such Form A or Form D crystal forms.

5

18. A method according to claim 17, wherein said glycogen phosphorylase dependent disease or condition is selected from the group consisting of hypercholesterolemia, hyperglycemia, hyperinsulinemia, hyperlipidemia, hypertension, atherosclerosis, diabetes, diabetic cardiomyopathy, infection, tissue ischemia, myocardial ischemia, and tumor growth.

10